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(54) **Titanium dioxide suspension for use in film coating.**

(57) The invention is directed to a pigment suspension, for use in film coating, comprising titanium dioxid , xanthan gum, and water. The pigment suspension does not settle or harden over an extended period of time, and may be used in the production of a film-forming pigment solution for coating pharmaceutical or veterinary tablets or confectionary pieces.

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TITANIUM DIOXIDE SUSPENSION FOR USE IN FILM COATING

The present invention relates to a titanium dioxide pigment suspension.

Pigment suspensions can be used for producing a film coating on such items as pharmaceutical or veterinary tablets and confectionary pieces. The pigment suspension is typically stirred into a larger volume of polymer solution. The resulting film-forming suspension is used in the coating process. The film coating, in the form of a very thin film, should be uniform and consistent from one batch of tablets to the next.

The technique of film coating is generally known in the prior art. U.S Patent No. 2,954,323 to Endicott et al. discloses the increased efficiency and superior coating properties obtained with film coating in general as compared to other processes of coating.

The present invention relates to a pigemnt suspension which comprises, as pigment, titanium dioxide. Such pigment suspensions for use in film coating are preferably sold having a concentration of titanium dioxide as high as possible. However, as the concentration of titanium dioxide increases, the suspension tends to become more viscous and may reach a point where it becomes difficult to pour from its container. Upon aging, a thick suspension of titanium dioxide may harden to the extent of becoming unusable.

In developing a high concentration pigment suspension, it is desirable to obtain a product in which th titanium dioxide particles form a stable suspension and will not settle for a prolonged period of time. The need is for a pigment suspension which will readily pour from its container and will maintain its uniform properties, during both transportation and storage, until ready for application in film coating.

U.S Patent No. 3,981,984 to Signorino discloses a pigment suspension which claims to achieve a high concentration of titanium dioxide in a non-aqueous solvent. This pigment suspension consists of titanium dioxide particles, a protective colloid such as hydroxypropyl cellulose, and a non-aqueous solvent such as ethanol. Signorino teaches that as the titanium dioxide particles are added to the solvent, the mixture becomes too viscous, and the further addition of the protective colloid serves to suspend the particles and reduce the viscosity.

In view of the increasingly strict requirements of governmental regulations in regard to the use of organic solvents, it has become desirable to obtain aqueous pigment suspensions. However, a high content of titanium dioxide in water is not normally possible for use in film-coating. Although titanium dioxide suspensions in an aqueous sugar syrup are known, such suspensions are not generally suitable for use in a film-forming polymer solution.

The present invention involved a search for a combination of ingredients which would permit a high content of titanium dioxide particles in an aqueous suspension useful in film coating. Due to the fact that the composition may comprise merely water and a very small amount of xanthan gum, not requiring the presence of organic solvents, the composition is very simple, safe and inexpensive to make.

The present invention aims to provide a high concentration pigment suspension which does not settle upon aging and a pigment suspension with a high titanium dioxide concentration which is capable of being transported to customers in containers, and which may readily be combined with a film-forming polymer solution by stirring as well as being non-flammable and non-hazardous during handling and safe for use in edible products intended for human consumption.

The pigment suspension of the present invention comprises a mixture of titanium dioxide, xanthan gum, and water. The present invention is further described in the following detailed description of preferred embodiments of the invention.

The titanium dioxide pigment employed in the present invention is preferably water dispersable titanium dioxide 3328, sold by Whitaker, Clarke and Daniels of South Plainfield, New Jersey. The pigment is suitably present in an amount by weight of about 20 to 75 percent, and most preferably in an amount by weight of about 30 to 60 percent.

Titanium dioxide is a relatively heavy pigment which, when mixed in a solvent, tends to settle out and form a thick non-pourable layer of pigment on the bottom of the container. It has now been found that an excellent aqueous pigment dispersion can be obtained by the addition of a very small amount of xanthan gum.

Xanthan gum is a high molecular weight polysaccharride produced in a fermentation process by the microorganism Xanthomonas campestris. The gum, which is produced as an exocellular coating surrounding the cell wall of the microorganism, is unique and very specific, and the properties thereof are constant and reproducable under given conditions.

Xanthan gum is known as a suspending or dispersing agent in various applications. For example, xanthan gum has been used to suspend solids in ceramic glazes, paints, and textile print pastes.

The use of xanthan gum to create a high concentration titanium dioxide pigment suspension for use in a film-forming process in the food and drug industry is believed to be entirely new.

A commercially available xanthan gum, suitable in the present invention, is KELTROL, and especially Keltrol F, a finely meshed xanthan gum, manufactured by Kelco, a division of Merk & Co., Incorporated.

The suspension of the present invention differs from other suspensions in that it can exhibit gel like behaviour or very fluid behaviour. Typically, the suspension actually sets up and only breaks down into a liquid by shearing action, such a produced by merely shaking the container of the pigment suspension, resulting a readily pourable pigment suspension.

The xanthan gum is generally present in the invention in amounts, by weight, ranging from 0.005 to 5 percent. As will be evident, typically only relatively very small quantities of the xanthan gum need be present in the suspension. A preferred range is from 0.05 to 0.5 percent.

Compositions of the present invention were tested by what is referred to as an oven test. An oven test is an accelerated method of assessing the long-term properties of a pigment suspension. The oven test typically involved heating the pigment suspension at 104°F for a period, initially of 96 hours. This accelerated test is believed to be equivalent to 3 to 4 months at 85°F. Compositions of the present invention have withstood heating at 104°F for one month. The oven test results were evaluated according to the following system.

RATING SYSTEM

1.0 A rock hard or very hard settle is obtained. The suspension fails to redisperse.

2.0 A paste or semi-hard solid is obtained. The suspension fails to pour from its container without force or requires the use of a spatula.

3.0 A threshold suspension, with some supernatant, but stable. After agitation, the suspension is still thick, but pourable.

4.0 A suspension with or without supernatant but no settle is obtained. The consistency is like thick yogurt. On agitation the suspension become fluid.

5.0 A soft, fluid dispersion with no settle is obtained. It pours from its container with no agitation and flows freely.

5.5 The suspension has no settle, but is very watery.

6.0 The suspension is too watery, and is not acceptable.

Example 1

In a blender, the following components were weighed out and mixed:

Component	Percent by Weight
Water	69.80%
Xanthan Gum	0.20%
TiO_2	30.00%

The xanthan gum was added to the water while mixing at a moderate speed. Mixing was maintained for about 3 minutes or until all of the gum had dissolved. The titanium dioxide was added slowly while the blender was mixing. The speed was adjusted to maintain a vortex in the mixture.

An oven test was performed at 104°F for 96 hours. A rating of 5.5 was obtained. After a further period of 21 days at 104°F, a rating of 5.5 was obtained, indicating that the properties of the pigment suspension remained stable.

Xanthan gum has unique properties which permit the creation of a stable and pourable titanium dioxide pigment suspension. Other gums or colloids, natural and synthetic, do not produce a satisfactory product. The following table illustrates other gums which were tried, but found unacceptable.

Table A

Component	Trial/ Percent Weight 1	2	3
Distilled Water	49.80	49.80	49.80
Titanium dioxide	50.00	50.00	50.00
Guar gum	0.20		
Polyvinylpyrrolidone		0.20	
KLUCEL			0.20
Rating	1.0	1.0	1.0

As shown in Table A, guar gum, polyvinyl-pyrrolidone, and KLUCEL, a brand of hydroxypropyl cellulose manufactured by Hercules Co. in Wilmington, Delaware, were unacceptable, resulting in a suspension that was immediately unusable. Gum arabic was satisfactory only at high levels such as 15 percent, too high for commercial practicability.

Conventional additives may be included in the present composition, as will be understood by those skilled in the art. For example, about 0.1 percent of an antimicrobial agent such a methylpropyl paraben or potassium sorbate is suitable.

It is to be understood that the foregoing detailed description and preferred embodiment are merely given by way of illustration, and modifications may be made within the scope of the invention.

Claims

1. A pigment composition comprising titanium dioxide, xanthan gum and water.

2. A composition as claimed in claim 1 wherein the titanium dioxide is present at about 20 to 75 percent by weight, the xanthan gum at about 0.005 to 5 percent by weight and the water at about 40 to 80 percent by weight.

3. A composition as claimed in claim 1 wherein the titanium dioxide is present at about 30 to 60 percent by weight, preferably about 50 percent, and the xanthan gum from about 0.05 to 1 percent by weight, preferably from 0.02 to 0.5 percent.

4. A composition as claimed in any one of claims 1 to 3 which further comprises an antimicrobial agent.

5. A composition as claimed in claim 4 wherein the antimicrobial agent is methylpropyl paraben or potassium sorbate.

6. A composition as claimed in claim 4 or claim 5 wherein the antimicrobial agent is present at about 0.1 percent.

7. A film-forming suspension which comprises a composition as claimed in any one of claims 1 to 6 in admixture with a polymer solution.

8. The use of a film-forming suspension as claimed in claim 7 in the coating of pharmaceutical or veterinary tablets and confectionary pieces or other foodstuffs.

9. A pharmaceutical or a veterinary tablet or a confectionary piece or other foodstuff whenever coated with a film-forming suspension as claimed in claim 7.

10. A process for the production of a composition as claimed in claim 1 which comprises mixing together xanthan gum and water, and then adding titanium dioxide, wherein the amounts of xanthan gum, water and titanium dioxide may be as further defined in claim 2 or claim 3.

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EUROPEAN SEARCH REPORT

Application number

EP 85 30 8939

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int Cl 4)
X	CA-A- 935 255 (CANADIAN TITANIUM PIGMENTS LTD.) * Page 4, lines 7-18; claims 1,5,8 *	1-4,6-10	A 61 K 9/34 C 09 D 17/00
Y	---	1,7-9	
A	DE-A-3 004 442 (HOECHST AG) * Claims; page 4, lines 3-26 *	1-10	
D,Y	--- US-A-3 981 984 (C.A. SIGNORINO) * Claims *	1,7-9	
X	--- US-A-3 663 284 (D.J. STANCLOFF) * Column 2, lines 18-26; column 3, lines 29-31; claims *	1-3,8-10	
	-----		TECHNICAL FIELDS SEARCHED (Int. Cl.4)
			A 61 K C 09 D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	19-08-1986	BERTE M.J.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
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